IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)
)
Takanori SHINOKI et al.)
)
Serial No.: 09/529,255   )
)   Group Art Unit:1771
Filed: April 11, 2000   )
)   Examiner: Terrel Morris
SUPPORT MEMBER FOR   )
SEMIPERMEABLE MEMBRANE   )

DECLARATION PURSUANT TO 37 C.F.R. §1.132

Honorable Commissioner of Patents and Trademarks
Alexandria, VA 22313-1450

Sir:

I, Takanori SHINOKI, declare that:

1. I am one of the co-inventors of the invention disclosed and claimed in the application identified in caption.

2. I currently reside at 2-34 Uguisudai Kawanishi-Shi, Hyogo-Ken, 666-0133 Japan.

3. I graduated the engineering department of Kyoto University, Master course of Fiber Chemistry on March 31, 1958.

4. I have been employed since May 1, 1995 by Miki Tokusyu Paper MFG. Co., Ltd., the assignee of the present application, and have been engaged in research and development of new product until the present.

5. I conducted the experiments in order to show that the closest prior art does not have a maximum pore size of 42 μm or less and to show that a " heat shrinkage stress at 200 °C of 0.1 - 0.6 g/d" and a " mean value of breaking length at an elongation of 5%" are critical to achieve the desired maximum pore size, and prepared the data in attached Exhibit A and Exhibit B.

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6.   It is declared by the undersigned that all statements made herein of undersigned's own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonments, or both, under 18 U.S. Code 1001 and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

Dated this   2 1   day of November, 2006

Takanori Shinoki
Takanori SHINOKI

Exhibit A

According to the method described in the present specification (line 18, page 31 to line 13, page 32) , polyethylene terephthalate stretched fibers (fineness x length=2.0-2.3 dtex (1.8 - 2.07 denier) x 5mm)having a specified double refraction (Δn) and heat shrinkage stress (200°C)shown in Table 1 below were prepared. The resultant fibers (60 parts by weight) and 40 parts by weight of polyethylene terephthalate unstretched fibers (Δn=0.012, fineness x length=1.2 dtex x 5mm) were put into the pulper. Then, according to the method described in the present specification (line 14, page 32 to line 10, page 33) , a paper was made and subjected to the heat calender processing to give nonwoven fabrics.

The physical properties (mean value of breaking length at an elongation of 5%, heat shrinkage stress (200°C) and maximum pore diameter) of the resultant nonwoven fabrics are shown in Table 1 below.

The mean value of breaking length at an elongation of 5% were measured according to the method described in the present specification (line 4 to line 21, page 30) .

The distribution of pore size was measured by COULTER POROMETER II (made by COULTER ELECTRON LTD.) . It is to be noted that the result obtained above is in good conformity with the bubble point method described on line 8-17 in page 31 of the present specification.

The distribution charts of pore diameter of Examples 1 and 3 and Comparative Example 1 are attached herewith as Exhibit B.

Table 1

	Polyester fiber		Physical properties of nonwoven fabric	
	Double refraction	Heat shrinkage stress (200°C)	Mean value of breaking length (5%)	Maximum pore diameter
	Δn	g/d	km	μm
Example 1	0.178	0.14	5.8	36
Example 2	0.184	0.29	6.2	37
Example 3	0.189	0.38	6.4	35
Example 4	0.193	0.40	6.6	36
Example 5	0.195	0.46	6.9	38
Example 6	0.199	0.51	7.1	32
Comparative Example 1	0.166	0.06	3.8	64
Comparative Example 2	0.178	0.08	3.3	71
Comparative Example 3 (Sinjou et al.)	USP 4,795,559		3.4	44

The support members in Examples 1 to 6 in Table 1 have respectively 5.0 km or more in mean value of breaking length at an elongation of 5% and 30 μm order in pore size (maximum pore diameter).

To the contrary, in Comparative Example 1, both the mean value of breaking length at an elongation of 5% and maximum pore diameter are low and the maximum pore diameter is such high as 64 μm. Comparative Example 1 is a typical example wherein the fiber used is the conventional one for use of clothing or industry.

Comparative Example 2 is an example wherein the fiber used is the one which was stretched to have a Δn value within the range as specified by the present invention but have a heat shrinkage stress (200°C) without the range as specified by the present invention, being caused by severe heat treatment. Such a fiber as in Comparative Example 2 is an example of

fiber used for reinforcing fiber for rubber such as tire cord
and conveyor belt.

Comparative Example 3 is to compare the present invention
with the closest prior art Shinjou et al. (U.S.P. No. 4795559).
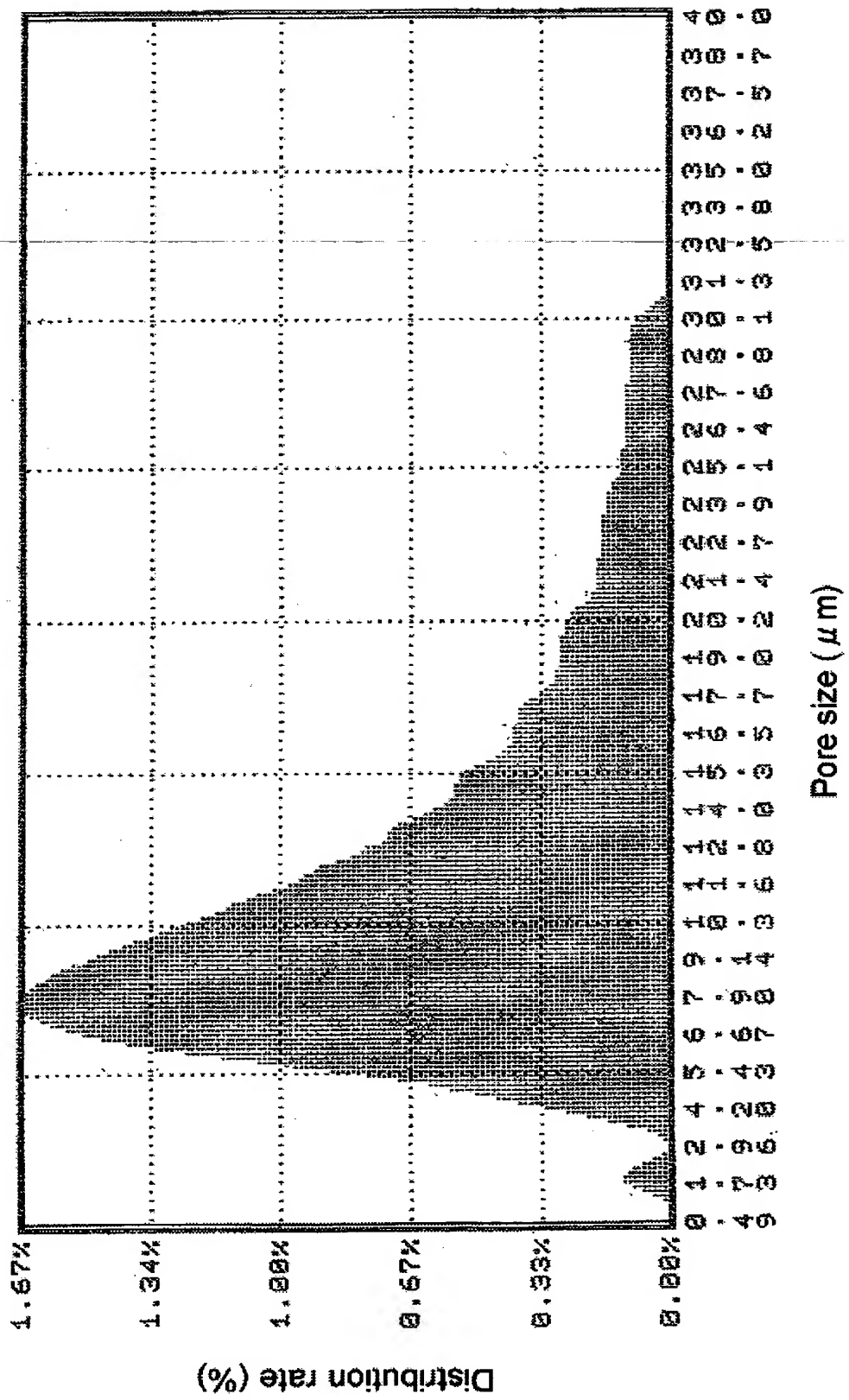The Shinjou et al. correspond to Japanese Patent Publication
No. 35009/1993 quoted in the present specification as one of
the closest prior arts.

With respect to Shinjou et al., DECLARATION PURSUANT TO
37 C.F.R. §1.132 was submitted to show advantages or unexpected
results of the present invention in comparison with Shinjou
et al. at the time of response to the official action issued
on November 16, 2004. The values of Comparative Example 3 in
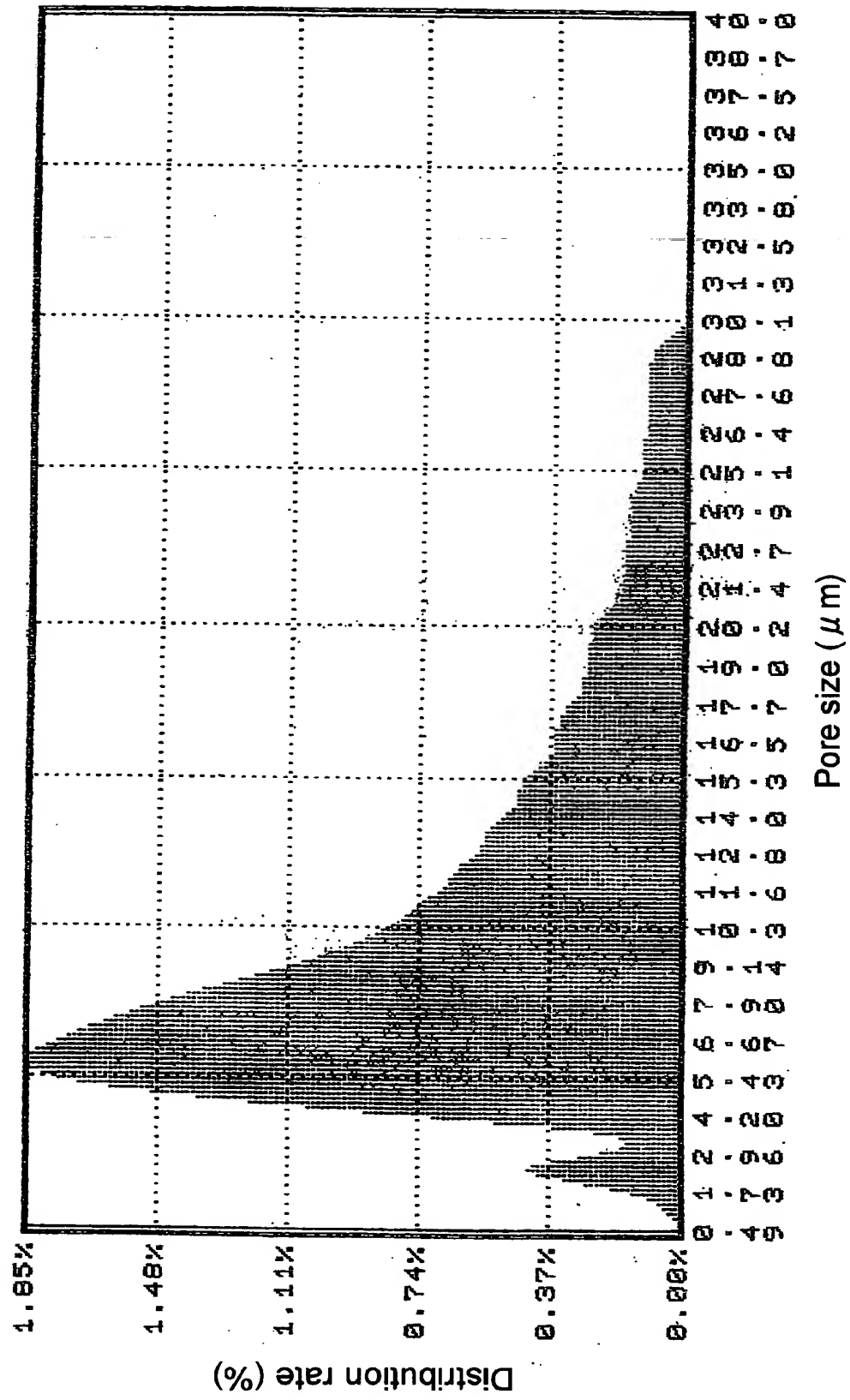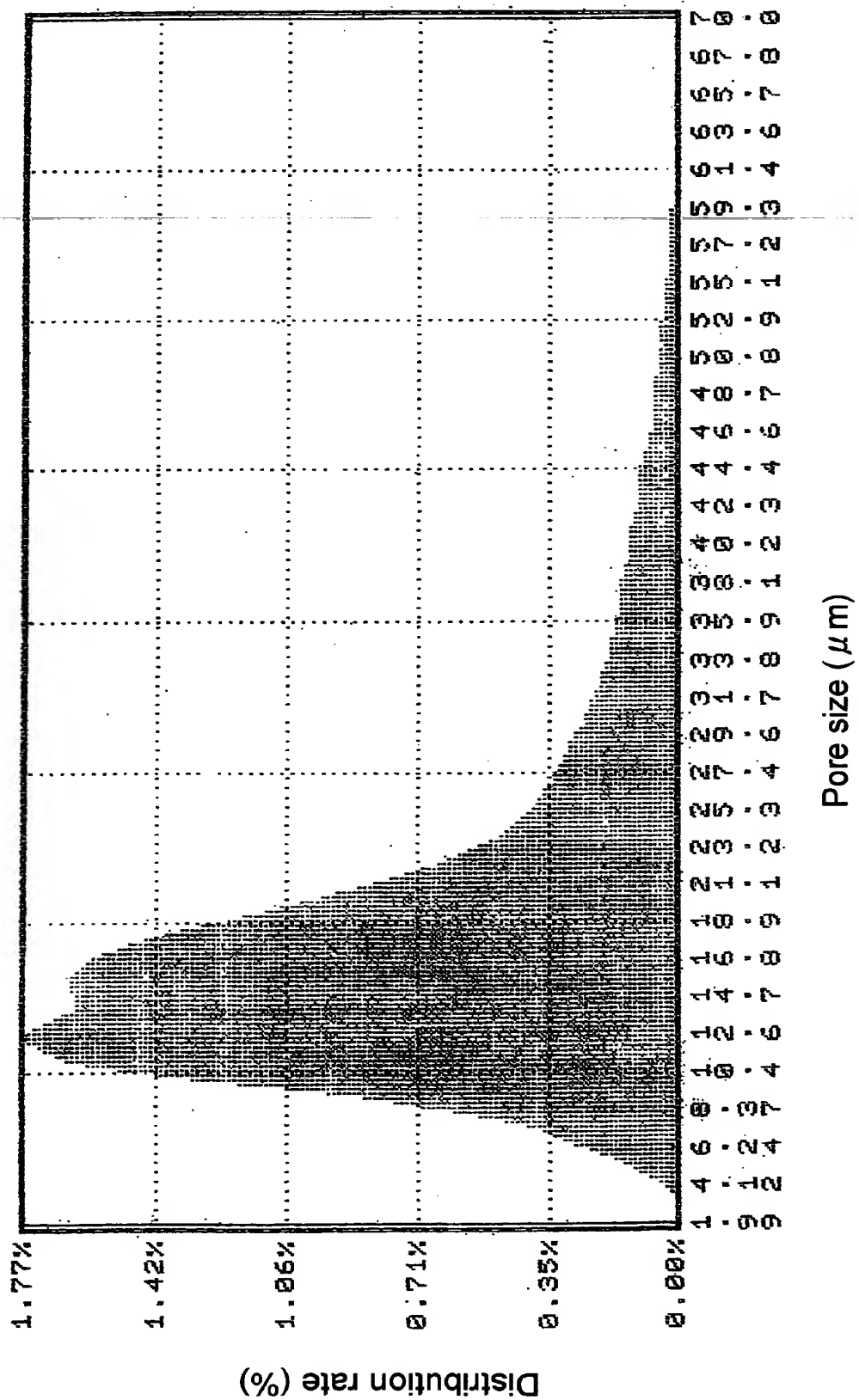Table 1 are the ones obtained in this DECLARATION.

END

Example 1

Distribution rate (%)

Pore size (μm)

Example 3

Distribution rate (%) vs Pore size (μm)

Comparative Example 1

Distribution rate (%) vs Pore size (μm)

Appendix

[Introduction]
In the OA [dated on 2006.7.12], the USPTO examiner discusses the patent application only by focusing on the width, kind, length, and combination of the fibers constituting the support, without looking at the properties of the support which are the most distinctive characteristic of the application. Apparently considering that the product of the present invention can be obtained from any raw materials, the examiner does not recognize new functions provided by combination of these factors and modification of the processing condition. He/she completely neglects the fact that the object of the present patent application, i.e., the reverse osmosis (RO) membrane support withstanding high-pressure filtration is possibly the world's first innovative technology. For more detailed understanding by the examiner, the present rebuttal is divided into two parts, and the "basic concept" of the technology in the present patent application will be described in the first part and specific rebuts to this OA in the paragraphs of second part below.

[Specific explanation]
I. Basic concept of the technology of present patent application
Production of ultrapure water by using RO membrane filter is practiced widely and thus, not particularly new. However, most of the ultrapure water is used only in applications demanding no particularly high pressure, such as semiconductor washing water, medical and pharmaceutical water, and food-related water, where purified water such as tap water is used as raw water. For desalination of sea water having a high osmotic pressure, used are methods extremely lower in productivity, such as distillation low in energy effectiveness and operation of RO membrane filter at a relatively lower pressure.

However, in the current increasing worldwide demand for pure water, there exists an urgent need for a method of producing ultrapure water by using a simple RO membrane filter.

(1) Requirements for a semipermeable membrane support withstanding high-pressure filtration seem to be:
① uniform distribution in size of pores in the nonwoven fabric support/uniform distribution of high pressure, and
② high dimensional stability causing almost no deformation during continuous long-term high-pressure filtration.

(2) Form and reasons of trouble common to conventional supports
[Pore size distribution]
As previously described in the written reply (with supplementary tests) concerning Shinjo patent (USP No. 4,795,559), there emerges sudden isolated large-sized pores in the micropore distribution curve. The term "isolated pores" mean pores that emerges as a sudden uncontinuous peak of large pores

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independently in the pore-size distribution curve of a support main body plotted from the lower pore-size side. As described several times previously, it is not possible to obtain a high-pressure-filtration filter support without eliminating such larger pores.

[Reasons for generation of isolated large-sized pores]
In conventional methods concerning support nonwoven fabrics, a blended sheet of two or more fibers substantially different in fineness is used as a means to control pore size. Wet-process nonwoven fabric is usually produced by binding at least 500,000 or more monofilaments (in the case of a monofilament of 2.0 denier) and cutting it into a predetermined length during processing, which inevitably causes adhesion between oil-coated monofilaments. Worse enough, polyethylene terephthalate fiber is hydrophobic and extremely lower in compatibility with water, and thus, PET staple fibers cannot be dispersed favorably even if they are added to and agitated in water by using a pulper. Coating of hydrophilic oil on the fiber surface normally leads to improvement of dispersion only to some extent. Increase in the amount of the oil coated only results in bubbling by the oil and deterioration in dispersibility. The phenomenon is an inevitable fact observed not only in preparation of such supports but also in preparation of conventional wet polyester nonwoven fabrics in general. Observation under electron microscope often reveals aggregates of 5 to 10 monofilaments bound to each other in parallel. Currently when the ideal monofilament dispersion is not achieved in production equipment, it is no use in propsing use of monofilaments having a diameter of 3.0 deniers or less as in G patent, if it is not possible to prevent generation of extremely thick fiber aggregates of bound fibers. These are the reasons for generation of the isolated large-sized pores.

[5% expansion stress]
The physical parameter is first proposed by the inventors as a parameter of nonwoven fabric. It may be probably because there was no prior literature at all discussing the dimensional stable support withstanding high-pressure filtration although there are many patents on RO membrane filter support. The 5% expansion stress in G patent (USP No. 5,851,355) estimated from the data (Table 1) therein is only about 20% of that in the present invention, indicating that the nonwoven fabric is still far from dimensionally stable.

For stabilization of the dimension of nonwoven fabric, it is needed to improve adhesion among fibers and thus, to use a fiber having a crystallinity as low as possible. However, it is also needed to use a fiber having a crystallinity as high as possible as its component, for providing the resulting nonwoven fabric with stability. The method of satisfying two conflicting properties, low and high crystallization, of the raw fiber as described above cannot be developed easily by any one even with the presence of such fibers as the examiner indicates. Details will be described below.

For convenience in description, patents on the support not taking high-pressure filtration into consideration will be referred to as "first-generation" patents, while those aimed at high-pressure filtration as "second-generation" patents. To be short, conventional patents belong to the "first-generation" patents, while the present patent application to the "second-generation" patent. Indiscriminative discussion on patents different in its object only leads to confusion. Hereinafter, major aspects of the present patent application will be described.

(3) Points of present invention
[Support withstanding high-pressure filtration: (1)uniform pore-size distribution]
As described above, because it is inevitable that the bound fiber is generated in wet polyester nonwoven fabric, the inventors considered that it was not possible to obtain uniform pore size distribution no matter how deeply the study was continued in this direction. Instead, the inventors found to form pores around monofilaments by providing respective monofilaments with high shrinkage force and allowing them to shrink simultaneously by heat during calendering. Because fine pores are formed by shrinkage of respective monofilaments, the pores are formed in the aggregates of bound fibers. Studies revealed disappearance of the large-sized pores observed in the first-generation patents.

The means is omitted here, as it is described in detail in the patent application, but it is necessary to raise orientation degree (Δn) of fiber as much as possible for obtaining a product with higher crystallinity. The fiber is heat-fused by calendering, and, because the fiber is preferably lower in crystallinity and softer for better fusion, the fiber was treated at a lower heat-set temperature after drawing at high magnification, for preservation of low crystallinity. Because a high-orientation fiber crystallizes even at room temperature, care should be given to prevent crystallization of the fiber and the conditions of storage and transportation are optimized.

It is possible to fuse such a high-orientation low-temperature-heat-set fiber favorably in heat-calendering step. The heat also accelerates crystallization of the high-orientation fiber, giving a nonwoven fabric of fibers having an extremely high crystallinity. Thus, it is possible to obtain a nonwoven fabric higher in tensile strength and Young's modulus.

The "heat shrinkage stress (g/d) at 200°C" is an indicator of the crystallization degree, and, currently in Japan, there is no wet-nonwoven-fabric manufacture providing such a raw fiber product with the value (dry heat shrinkage stress at 200°C). It is because there is no need for it in Japan, and the same is probably true in the U.S. The physical parameter is also proposed by the inventors.

[Support withstanding high-pressure filtration: (2) high 5% expansion stress]
The high-orientation low-heat-set fiber has favorable calendering efficiency and has high interfiber adhesive strength. In addition, it is possible to obtain a product highly favorable in dimensional stability, by accelerating crystallization

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of the respective monofilaments constituting the nonwoven fabric in the calendering step. High orientation is effective in accelerating crystallization. The most effective fiber property is the orientation degree (Δn), although it is essential to determine calendering conditions and subsequent heat history carefully, because they also exert great influences.

The breaking length of 4.0 km or more was decided, in collaboration with a RO membrane filter manufacturer currently performing seawater desalination at a high pressure of 100 kg/cm2, and included in the acceptance specification of the inventors' company, and thus, it is not an arbitrary physical parameter by any means. The dimensional stability of a support practically governs the lifetime of the support, and thus, there is a severe user requirement imposed thereon.

(4) First- and second-generation technologies
The nonwoven fabric in the Shinjo patent (USP 4,795,559), which was previously cited by the examiner as a prior art of the present patent application, was a laminate of dry and wet nonwoven fabrics, but, as it is widely understood, the pore size of dry nonwoven fabrics is far larger than that of wet nonwoven fabrics, and the nonwoven fabric having the pore size distribution curve attached to our written reply was a wet nonwoven fabric. There are several support nonwoven fabrics commercially available in Japan in addition to that described above, each of these nonwoven fabrics has a pore size distribution curve in a pattern similar to that of the Shinjo patent.

The original application of Shinjo patent (USP '559) was filed in Japan (JP-B No. 5-35009), and the inventors are well aware of the application and cited the prior art in our present patent application (P.4 L.21 to P.5 L.8 of the present patent application) and describ there that the present application overcame the disadvantages in this point. It is also described that the inventive nonwoven fabric support is a support having properties not found in that in Shinjo patent, although the term "second generation" was not used. Alternatively, G patent (USP '355) cites the Shinjo patent at great length in its introductory region ("BACKGROUND OF THE INVENTION") and describes the "basic concept" behind the G patent technology (first column, L.45 to second column, L.31).

According to the G patent, the G patent provides a 100% wet nonwoven fabric, in contrast to the two-layered (dry and wet) nonwoven fabric in the Shinjo patents and others. In the G patent there is no second-generation-based idea at all, in pointing out and overcoming the problems in properties in the Shinjo patent. Thus, the G patent is a typical first-generation application technically as described above, and the fact is even recognized even by Goettmann himself.

Citation of first-generation data as the prior art of the present second-generation application by the examiner is based on the fact that the examiner does not understand the technical background and the "basic concept" of the present patent application at all. Accordingly, the best way to eradicate the doubt of

examiner would be to prepare a sample according to the G patent, measure the pore size distribution curve, and determine the presence or absence of the isolated pores. However, it is not possible to reproduce the G patent.

II. Opinions to current OA (by paragraph)

<OA P3 L.1 to 14>
(1) The blending rate of the polyester fibers for the support was described there at great length, but the data based on a technical "basic concept" different from that of the patent application do not make sense as OA, no matter how lengthy they are. Prior art indicating that the second-generation support was prepared should be cited.

(2) The "Frazier air permeability" was explained several times in the past. However, the same comment continues. The air permeability (by Frazier method) is only an average, and may constitute a necessary condition but not a sufficient condition in filter applications. We believe it is too rough to discuss support properties with the value. Details will be described with examples.

For example, there are two kinds of support having 10 pores in the same area. One of them (A) has holes having pore sizes gradually increasing from 1 µm to 10 µm stepwise by 1 µm. The other (B) has 10 pores having the same diameter of 5.5 µm. Both supports A and B have an average pore size of 5.5 µm. According to the examiner, the filter properties should be the same between these supports, because the averages are the same. However, care should be given to the fact that the filtering properties are completely different. Most of fluid flows into the pores of 10 µm or 9 µm in support A. The other eight pores do not participate in filtration. The support and the semipermeable membrane resin are separated easily, because the membrane resin does not flow into the pores of 1 to 2 µm in diameter well, and thus, the adhesive strength between substrate and resin is practically none. The liquid flows through all 10 pores in the support B, and thus, the filtration area is very large, allowing stabilized operation. Of course, the adhesive strength between support and resin is sufficiently high.

<OA P.3, L.16 to 19>
As described above, in the present patent application aimed at producing a second-generation support, all monofilaments are highly oriented and heat-set at low-temperature, and then calendered. The monofilaments shrink rapidly to a certain amount, are converted into high-crystallinity filaments by crystallization occurring simultaneously, and become resistant to deformation and stabilized

dimensionally rapidly. (Such a phenomenon is called "restricted shrinkage" by those skilled in the art of fiber). The condition causing "restricted shrinkage" of all fibers in the support nonwoven fabric is a physical parameter, " heat shrinkage stress at 200°C (g/d)", which is probably proposed by the inventors. Care should be given to the fact that it is completely different from the dry heat shrinkage percentage (%) commonly used as an indicator of the property of textile fiber. The dry heat shrinkage stress at 200°C of common commercially available textile polyethylene terephthalate fibers is about 0.04 to 0.06 (g/d).

<OA P.3, L.20 to 22>
[Heat calendering described in G patent]
As repeatedly explaining in the written reply previously submitted, it is not possible to prepare even a single sample because of thermal fusion and adherence of the web onto roll, when the web is heat-calendered at a temperature higher than the melting point of the binder fiber. Note the fact that the web was heated only up to 215°C (binder fiber melting point: 267°C) for bonding dry and wet polyethylene terephthalate fiber webs in the Shinjo patent (USP '559, Column 6, L.6 to L.24). The Goettmann proposal of the calendering of a wet nonwoven fabric web at a temperature of not lower than the melting point of binder fiber is probably the first in the world. The examiner gives no information about possibility of reproduction of Goettmann product although the examiner clings to the Goettmann prior art and thus.
<OA P.4, L.1 to 10>
As described above, a semipermeable membrane support withstanding high-pressure filtration should have a pore size distribution as uniform as possible. The first-generation supports having isolated larger-diameter pores as described in the Shinjo patent cannot be applied to high-pressure filtration. The phrase "pore size (maximum pore size) up to 42 µm" in the present patent application indicates extreme evenness of the pore size distribution. Unfortunately, we did not have a porometer (pore size distribution analyzer) allowing accurately measurement before application of the present invention, and thus, used the pore size instead. The argument in this section would be understandable, with reference to the porometer chart submitted.

It is quite natural that the G patent has no description on pore size, because it only considers the first-generation support as described above.

<OA P.4, L.11 to P.5, L.4>
The current most annoying problem for us is that it is not possible to reproduce the G patent product. If such a sample is prepared, the inventors are ready to determine the pore size distribution curve immediately and eradiate the doubt of the examiner. We have reported several times that we found difficulty in sheeting and calendering, in repeating the tests described in the G patent. The examiner's comment that "the patent is valid" does not constitute a reply. Let us know specifically in which region of the G patent there is such description on experimental method. We are expecting the examiner's reply, which lead to

temporary standstill.

Fortunately, the Shinjo patent, a target of the G patent, allows double checking, and the pore size distribution, 5% expansion stress, and interlayer adhesion strength with the semipermeable membrane resin were obtained. As described in the introduction of the present patent application, there are some disclosed patents on such a support in Japan, which allow double check thereof and measurement of pore size distribution curve. All of the supports show the isolated distribution. Because it is not possible to prevent binding of polyester monofilaments in water at the commercial production scale, it is possible to estimate the details and potential of the G patent roughly from these data.

<OA P.5, L.5 to 11>
Reasons based on the misunderstanding of the present patent application aimed at developing a second-generation support

There is difference in "basic concept" between the G patent and the present technology. Prior art aimed at developing a second-generation support should be cited.

When the element is amended to "(ii) subjecting the monolayered paper web to a heat treatment at a temperature lower the melting point of the binder resin under pressure to bind the fibers to each other," it can be argued that resultant products between the present invention and Goettmann are completely different.

<OA P.6, L.18 to P.7, L.7>
Observation under an electron microscope of the surface or cross section of a wet polyester-fiber nonwoven fabric, typically of polyethylene terephthalate, reveals aggregates of 5 to 10 monofilaments bound to each other. There is no exception. The aggregates are inevitable, if the nonwoven fabric is mass-produced in commercial scale. The bound fiber aggregates exert an adverse influence similar to that when extremely thick fibers are added on the first-generation support. The present patent application is completed, by facing and overcoming the severe fact. The problem is lack of such an image by the examiner.

<OA P.7 L.8 to 18>
We agree with the examiner's opinion that the scope of a patent is not restricted only to the region described. However, it should not be expanded, if the interpretation extends beyond our common sense or obvious natural phenomenon. Although described in the previous written reply, we would like to describe the problems that we cannot double check the G patent once again, because there is no reply from the examiner. Please let us know the opinion about the followings.

(1) Figure 2 of the G patent: the web 32 coming out of the paper machine 28 becomes in the "space walk" state without conveyor belt, before reaching the

drying roll 30 in the next step. We believe that is practically impossible on the earth where there is gravity, and please let us know what the opinion of the examiner is.

(2) Calendering temperature: calendering at a temperature not lower than the melting point of binder is unheard of. The web is wound around the heating roll. We have no intention to argue the patentability of G patent with USPTO. We simply want "Detailed Description" allowing the third party to double check easily, because the examiner cites the prior art.

<OA P.7, L.19 to P.8, L.17>
We believe that we can determine the pore size distribution curve by using a porometer immediately and show that the isolated distribution is inevitable in the G patent, if the reproduction of the G patent is possible.

[Summary]
The object of the present patent application is effective utilization of water resource by recycling sea water, salt water (lake water containing salts and others), and sewage (waste water).

As the secretary-general of the United Nations described in the UN environmental development summit held several years ago (in South Africa, Aug. to Sep. 2002), "there will be water shortage for 3.5 billion people, almost half of the world population, by 2025", and water regeneration and purification is an urgent problem at the worldwide level. It is quite important socially whether it is possible to perform high-pressure filtration by using RO membrane filter.

The present patent application was of course filed also in Japan and the patent was granted on 2001.1.26 (patent number: 3153487). We know that the present application shall be evaluated according to the criteria in the USPTO. However, please understand that the application is not a trivial application and we will continue to engage in development aimed at solving global environmental problems. We sincerely hope the examiner's careful examination.

END